

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PC 03/07116

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
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INTERNATIONAL SEARCH REPORT

Intern Application No
PCT 03/07116A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C08G63/80 C08G63/78

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C08G B01J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ, COMPENDEX

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	GB 1 190 801 A (EASTMAN KODAK CO) 6 May 1970 (1970-05-06) page 2, line 94 - line 126 page 3, line 99 - line 106 page 4, line 33 - line 57 page 5, line 1 - line 20 page 5, line 51 - line 88 ---	1, 27, 28, 31, 36
A	US 5 408 035 A (DUH BEN) 18 April 1995 (1995-04-18) cited in the application the whole document ---	1-55
A	US 3 953 404 A (BORMAN WILLEM F H) 27 April 1976 (1976-04-27) the whole document ---	1-55
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☒ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

* Special categories of cited documents:

- *A* document defining the general state of the art which is not considered to be of particular relevance
- *E* earlier document but published on or after the international filing date
- *L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- *O* document referring to an oral disclosure, use, exhibition or other means
- *P* document published prior to the international filing date but later than the priority date claimed

- *T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- *X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- *Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- *Z* document member of the same patent family

Date of the actual completion of the international search

16 October 2003

Date of mailing of the international search report

23/10/2003

Name and mailing address of the ISA

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INTERNATIONAL SEARCH REPORT

International Application No.

PCT/EP 03/07116

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 4 276 261 A (KERSCHER FREDERICK C ET AL) 30 June 1981 (1981-06-30) the whole document ----	1-55
A	US 4 370 302 A (OKUHIRA TOSHIFUMI ET AL) 25 January 1983 (1983-01-25) the whole document ----	1-55
A	EP 0 953 589 A (MITSUI CHEMICALS INC) 3 November 1999 (1999-11-03) paragraphs '0039!-'0048! ----	1-55
A	US 5 711 089 A (PIKUS ILYA) 27 January 1998 (1998-01-27) the whole document ----	1-55
A	EP 0 419 400 A (GOODYEAR TIRE & RUBBER) 27 March 1991 (1991-03-27) the whole document ----	1-55
X	US 3 075 952 A (COOVER JR HARRY W ET AL) 29 January 1963 (1963-01-29) column 1, line 38 -column 2, line 64 column 3, line 71 -column 4, line 23 column 5, line 15 - line 72 column 6, line 25 - line 33 -----	1,27,28, 36

PATENT COOPERATION TREATY

From the
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

PCT

To:

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ITALIE

NOTIFICATION OF TRANSMITTAL OF THE INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Rule 71.1)

Date of mailing
(day/month/year)

02.11.2004

Applicant's or agent's file reference
9497.01/PC

IMPORTANT NOTIFICATION

International application No.
PCT/EP 03/07116

International filing date (day/month/year)
03.07.2003

Priority date (day/month/year)
09.08.2002

Applicant
CAVAGLIA', Giuliano

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary examination report and its annexes, if any, established on the international application.
2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.
3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.

4. REMINDER

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices) (Article 39(1)) (see also the reminder sent by the International Bureau with Form PCT/IB/301).

Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

The applicant's attention is drawn to Article 33(5), which provides that the criteria of novelty, inventive step and industrial applicability described in Article 33(2) to (4) merely serve the purposes of international preliminary examination and that "any Contracting State may apply additional or different criteria for the purposes of deciding whether, in that State, the claimed inventions is patentable or not" (see also Article 27(5)). Such additional criteria may relate, for example, to exemptions from patentability, requirements for enabling disclosure, clarity and support for the claims.

Name and mailing address of the international
preliminary examining authority:



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PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)



Applicant's or agent's file reference 9497.01/PC	FOR FURTHER ACTION See Notification of Transmittal of International Preliminary Examination Report (Form PCT/PEA/416)	
International application No. PCT/EP 03/07116	International filing date (<i>day/month/year</i>) 03.07.2003	Priority date (<i>day/month/year</i>) 09.08.2002
International Patent Classification (IPC) or both national classification and IPC C08G63/80		
Applicant CAVAGLIA', Giuliano		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.
2. This REPORT consists of a total of 5 sheets, including this cover sheet.
- ☒ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of 7 sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the opinion
- II ☐ Priority
- III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☐ Certain defects in the international application
- VIII ☐ Certain observations on the international application

Date of submission of the demand 02.02.2004	Date of completion of this report 02.11.2004
Name and mailing address of the international preliminary examining authority:  European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized Officer Masson, P Telephone No. +49 89 2399-2051 

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. **PCT/EP 03/07116**

I. Basis of the report

1. With regard to the **elements** of the international application (*Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17)*):

Description, Pages

1-18 as originally filed

Claims, Numbers

8-52 received on 02.02.2004 with letter of 28.01.2004
1-7 filed with telefax on 12.07.2004

Drawings, Sheets

1/1 as originally filed

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
☐ the language of publication of the international application (under Rule 48.3(b)).
☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
☐ filed together with the international application in computer readable form.
☐ furnished subsequently to this Authority in written form.
☐ furnished subsequently to this Authority in computer readable form.
☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
☐ the claims, Nos.:
☐ the drawings, sheets:

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/EP 03/07116

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)).

(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

6. Additional observations, if necessary:

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes: Claims	1-52
	No: Claims	
Inventive step (IS)	Yes: Claims	
	No: Claims	1-52
Industrial applicability (IA)	Yes: Claims	1-52
	No: Claims	

2. Citations and explanations

see separate sheet

Cf V:

Reference is made to the following documents:

- D1: GB-A-1 190 801 (EASTMAN KODAK CO) 6 May 1970 (1970-05-06)
- D2: US-A-5 408 035 (DUH BEN) 18 April 1995 (1995-04-18)
- D3: US-A-3 953 404 (BORMAN WILLEM F H) 27 April 1976 (1976-04-27)
- D4: US-A-4 276 261 (KERSCHER FREDERICK C ET AL) 30 June 1981 (1981-06-30)
- D5: US-A-4 370 302 (OKUHIRA TOSHIFUMI ET AL) 25 January 1983 (1983-01-25)
- D6: EP-A-0 953 589 (MITSUI CHEMICALS INC) 3 November 1999 (1999-11-03)
- D7: US-A-5 711 089 (PIKUS ILYA) 27 January 1998 (1998-01-27)
- D8: EP-A-0 419 400 (GOODYEAR TIRE & RUBBER) 27 March 1991 (1991-03-27)
- D9: US-A-3 075 952 (COOVER JR HARRY W ET AL) 29 January 1963 (1963-01-29)

The document D10 (US-A-3 767 601) was not cited in the international search report. A copy of the document is appended hereto.

Article 33(2) PCT:

Claim 1 discloses a process for the solid phase continuous polymerisation of polyesters.

None of the documents discloses the same combination of features as disclosed in **claim 1**.

Thus, the subject matter of **claim 1** meets the requirements of Article 33(2) PCT.

The same considerations apply to the subject matter of **dependent claims 2 to 52** which contain merely conventional embodiments of **claim 1**.

Article 33(3) PCT:

Document D5 is considered as representing the closest prior art. The difference between **D5** and **claim 1** is that in **D5** the reactor is not inclined and furthermore, the solid state polymerization (SSP) takes place in vacuum (column 6; line 34). Since there are no comparative examples in accordance with **D5**, the objective technical problem is to provide further reactor for making SSP of polyester.

There is an indication in **document D10** to use a slight inclined rotary reactor for increasing the IV of polyester granules with a purging gas (column 1; lines 53 to 68; column 2; lines 43 to 52).

Thus, no inventive step should be acknowledged for the subject matter of **claim 1**.

Thus, the subject matter of **claim 1** does not meet the requirements of Article 33(3) PCT.

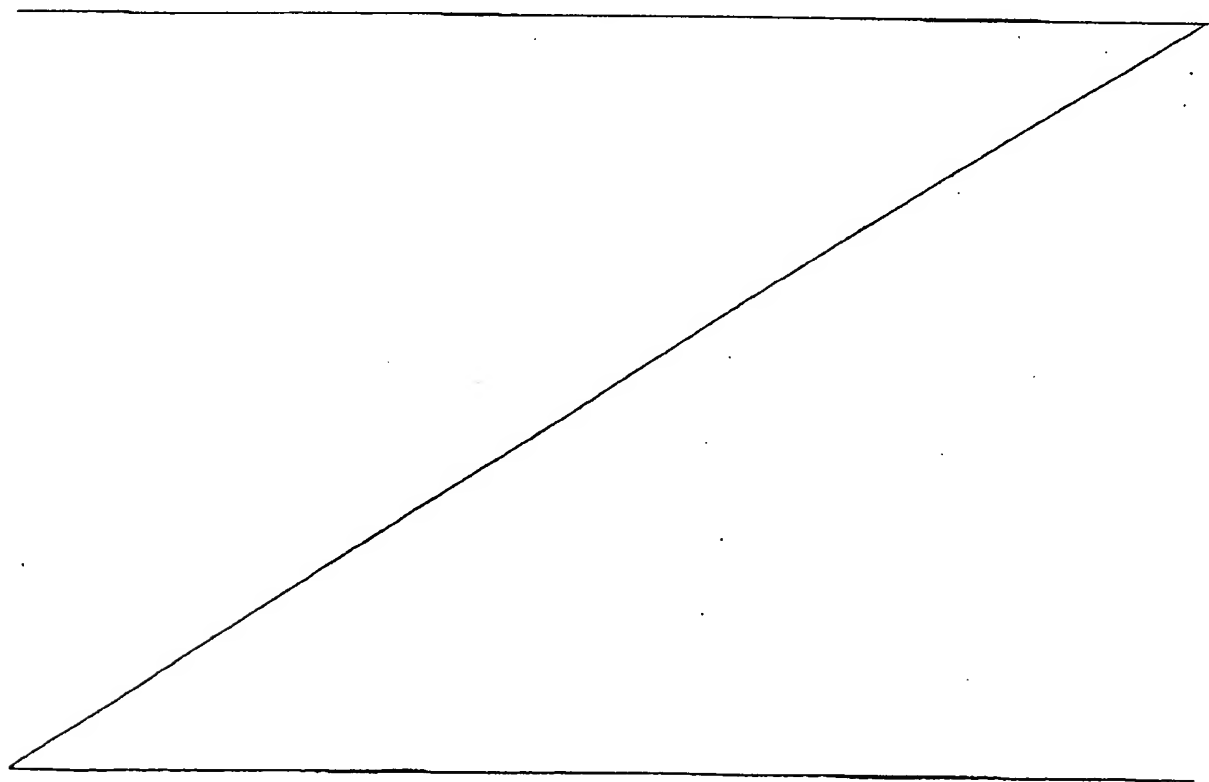
The same considerations apply to the subject matter of **claims 2 to 52** which contain merely conventional embodiments of **claim 1**.

Article 33(4) PCT:

The subject matter of **all claims** is capable of industrial applicability.

AMENDED CLAIMS

1. A process for the solid phase continuous polymerisation of polyesters, comprising the steps of:
- preparing a mass of polyester prepolymer granules, of at least one polyester;
 - 5 - feeding said polyester prepolymer granules to a crystalliser (13) where they are heated up to a suitable temperature to cause the crystallisation of the granules;
 - feeding said crystallised granules at a temperature comprised in the range 170°C ÷ 235°C into at least an horizontal, cylindrical, rotary reactor (15), said reactor being slightly inclined;
 - 10 - producing a purge gas flow inside said reactor (15);
 - causing the intrinsic viscosity (IV) increase of said at least one polyester by making said granules move forward through said reactor (15) thanks to its rotation and inclination.



9497.01

19 **A**~~REVENDED CLAIMS~~

~~1. A process for the solid phase continuous polymerisation of polyesters, comprising the steps of:~~

- preparing a mass of granules, crystallised and at high temperature, of at least a polyester, wherein said high temperature is comprised in the range $170^{\circ}\text{C} \div 235^{\circ}\text{C}$;
- feeding said granules into a plant comprising at least an horizontal, cylindrical, rotary reactor (15), said reactor being slightly inclined;
- producing a purge gas flow inside said reactor (15);
- causing the intrinsic viscosity (IV) increase of said at least one polyester by making said granules move forward through said reactor (15) thanks to its rotation and inclination.

2. A process according to claim 1, wherein the crystallisation degree X_c of said polyester granules fed into said reactor is comprised in the range of $0 \div 70\%$, and wherein said reactor (15) rotates at a speed comprised between 0,1 and 10 r.p.m., and wherein the angle of inclination (α) of said reactor with respect to the horizontal plane is comprised between 0.1 and $3,5^{\circ}$.

3. A process according to claim 1 or 2, wherein said polyester granules fed into said reactor have a temperature comprised in the range of $185 - 225^{\circ}\text{C}$.

4. A process according to claim 1 or 2, wherein said polyester granules fed into said reactor have a temperature comprised in the range of $180 - 230^{\circ}\text{C}$.

5. A process according to any claim from 1 to 4, wherein said polyester granules fed into said reactor have a crystallisation degree $X_c > 10\%$.

6. A process according to any claim from 1 to 4, wherein said polyester granules fed into said reactor have a crystallisation degree $X_c > 20\%$.

7. A process according to any claim from 1 to 4, wherein said polyester granules fed into said reactor have a crystallisation degree X_c comprised in the range of $0 \div 50\%$.

8. A process according to any preceding claim, wherein the rotation of said reactor occurs around its own central axis (S).
9. A process according to any claim from 1 to 8, wherein said reactor rotates at a speed comprised between 0,1 and 2,0 r.p.m..
- 5 10. A process according to any claim from 1 to 9, wherein the angle of inclination (α) of said reactor with respect to the horizontal plane is comprised between 3,0 and 12,0°.
11. A process according to any preceding claim, wherein downstream said reactor (15) at least a second horizontal, cylindrical, rotary, slightly inclined reactor is provided.
12. A process according to claim 11, wherein said granules move from one reactor to the
10 subsequent one by gravity.
13. A process according to claim 11, wherein the temperature of the polyester granules, subjected to the polymerisation passing through said reactors, is increased during the movement from one reactor to the subsequent of a value comprised between 2 and 20°C.
14. A process according to claim 11, wherein the temperature of the polyester granules
15 passing through said reactors is decreased from one reactor to the subsequent of a value comprised between 2 and 10°C.
15. A process according to claim 13, wherein the temperature of the polyester granules exiting one reactor is increased by means of an intermediate pre-heater located before the entrance into the subsequent reactor.
- 20 16. A process according to claim 11, wherein the polymerisation temperature in a first reactor is lower than the polymerisation temperature in a second reactor located downstream said first reactor.
17. A process according to claim 11, wherein the temperature of the polyester granules exiting one reactor is decreased by means of an intermediate cooler located before the
25 entrance into the subsequent reactor.

18. A process according to any claim from 1 to 17, wherein the ratio between the length of said reactor and its diameter of reactor is > 5 .
19. A process according to any preceding claim, wherein the ratio between the residual volume in said reactor inside which said granules have been fed, and its unloaded volume is $> 0,1$.
20. A process according to any preceding claim, wherein the flow regime of the polyester granules inside the reactor is characterised by a Froude Number $Fr = (\omega^2 \cdot R/g)$ comprised in the range of $1 \cdot 10^{-4} \div 0,5$, where ω is the angular velocity of the reactor; R is the internal radius of the reactor and g is the gravity acceleration = $9,806 \text{ m/s}^2$.
21. A process according to any preceding claim, wherein the temperature inside said at least one reactor is maintained at a constant value $\pm 10^\circ\text{C}$.
22. A process according to any preceding claim, wherein the internal diameter of said reactor is comprised between 0,5 and 10 meters.
23. A process according to any claim from 1 to 21, wherein the internal diameter of said reactor is comprised between 0,3 and 6 meters.
24. A process according to any preceding claim, wherein the flow of said purge gas in said reactor is conveyed in an opposite direction with respect to the flow direction of said granules that pass through said reactor.
25. A process according to any claim from 1 to 23, wherein the flow of said purge gas in said reactor is conveyed in the same direction with respect to the flow direction of said granules that pass through said reactor.
26. A process according to any preceding claim, wherein the ratio between the mass of the purge gas flow that passes through said reactor and the mass of the polyester granules in the reactor is $> 0,62$.
27. A process according to any preceding claim, wherein the ratio between the mass of the

purge gas flow that passes through said reactor and the mass of the polyester granules in the reactor is $> 0,9$.

28. A process according to any preceding claim, wherein said purge gas is an inert gas or air.

5 29. A process according to any claim from 1 to 27, wherein said purge gas is air with a dew point $< -30^{\circ}\text{C}$.

30. A process according to any claim from 1 to 27, wherein said purge gas is a mixture of gases chosen from the group comprising nitrogen, noble gases, carbon dioxide, carbon monoxide and oxygen and wherein the oxygen content is $< 10\%$ by weight.

10 31. A process according to any claim from 1 to 27, wherein said purge gas is a mixture of gases chosen from the group comprising nitrogen, noble gases, carbon dioxide, carbon monoxide and oxygen and wherein the oxygen content is $< 6\%$ by weight.

32. A process according to any preceding claim, wherein the purge gas is recycled to the reactor, after having been purified of the organic impurities, until a level of organic impurities
15 ≤ 100 p.p.m. by weight (CH_4 equivalent) has been reached.

33. A process according to any preceding claim, wherein said polyester is polyethylene terephthalate or PET.

34. A process according to claim 33, wherein said polyethylene terephthalate or PET has an IPA (Isophthalic Acid) content comprised in the range of $1 \div 20\%$.

20 35. A process according to claim 33 or 34, wherein said granules of polyethylene terephthalate fed into said reactor have an intrinsic viscosity comprised in the range between 0.55 and 0.65 dl/g.

36. A process according to claim 33 or 34, wherein said granules of polyethylene terephthalate fed into said reactor have an intrinsic viscosity comprised in the range between
25 0.25 and 0.75 dl/g.

37. A process according to any claim from 1 to 32, wherein said polyester is PEN polyethylene naphthalate.
38. A process according to any claim from 1 to 32, wherein said polyester is PBT polybutylene terephthalate.
- 5 39. A process according to any claim from 1 to 32, wherein said granules fed in the reactor have a carboxyl end groups content in the range of 10 ÷ 45%.
40. A process according to any preceding claim, wherein said granules are cube-shaped with dimensions comprised between 1 mm³ and 125 mm³.
41. A process according to any claim from 1 to 39, wherein said granules are spherical
10 with a diameter comprised between 1 mm and 5 mm.
42. A process according to any claim from 1 to 39, wherein said granules are extended cylinders of length < 10 mm and circular or square cross-section having, respectively, diameter and side < 5 mm.
43. A process according to any claim from 1 to 39, wherein said polyester granules are
15 pancake-like platelets of diameter > 3 mm and thickness < 3 mm.
44. A process according to any claim from 1 to 39, wherein said polyester granules have an irregular shape with a volume comprised between 1 and 125 mm³.
45. A process according to any preceding claim, wherein said mass of crystallised granules is achieved by subjecting the polyester granules to a crystallisation step in a fluidised-bed
20 crystalliser (13), said bed being fluidised by means of a gas flow sufficient to generate the fluidisation of the polyester granules with or without mechanical vibration.
46. A process according to claim 45, wherein said gases employed for the crystallisation are inert gases or air.
47. A process according to claim 45 or 46, wherein said crystallisation step is performed
25 with a residence time comprised between 2 and 20 minutes and, preferably, from 10 to 15

minutes.

48. A process according to claim 45, wherein the granules are heated to cause the crystallisation up to temperatures comprised between 140°C and 235°C and preferably in the range 200 – 225°C.

5 49. A process according to any preceding claim, wherein inside said reactor the polyester granules are subjected to a solid phase polycondensation and/or drying and/or crystallisation and/or dealdehydisation.

50. A process according to any preceding claim, wherein the intrinsic viscosity of the polyester is increased of at least 0,35 dl/g during the solid phase polymerisation.

10 51. A process according to any preceding claim, wherein the intrinsic viscosity of the polyethylene terephthalate is increased of at least 0,4 dl/g during the solid phase polymerisation.

52. A process according to any preceding claim, wherein said plant further comprises at least one vertical reactor located upstream and/or downstream said horizontal reactor (15).

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